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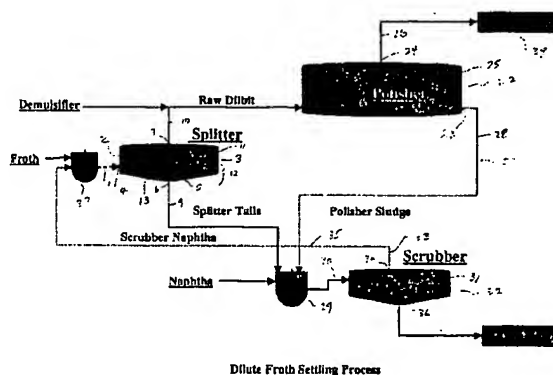
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(54) METHODE DE DECONTAMINATION ETAGEE POUR ELIMINER L'EAU ET LES SOLIDES PRESENTS DANS LA MOUSSE D'EXTRACTION DE SABLES BITUMEUX

(54) STAGED SETTLING PROCESS FOR REMOVING WATER AND SOLIDS FROM OIL SAND EXTRACTION FROTH

(57)

Diluent-diluted bitumen froth containing bitumen and diluent hydrocarbons, water, sand and fines (collectively "dilfroth") is fed into a gravity settler ('splitter') and temporarily retained to produce a bottom layer of tails comprising sand and middlings, a rag layer of discrete three-dimensional structures, each comprising hydrocarbons contained in a skin of fines, and a top layer of hydrocarbons containing small droplets of water and fines ('raw dilbit'). The flux in the splitter is less than 6 m³/h of dilfroth fed per m² of horizontal cross-sectional rag area. The in-coming dilfroth is fed directly into the splitter middlings. Demulsifier is added to the overflow stream of raw dilbit and the mixture is subjected to prolonged settling in a polisher tank, to produce polished dilbit containing less than 1.0% water and 0.3% solids. The splitter underflow tails, containing less than 15% bitumen, is mixed with diluent to raise the diluent/bitumen ratio to 4 to 10 and is gravity settled in a scrubber. Scrubber overflow, mostly diluent containing residual bitumen stripped from the tails, is recycled to the splitter. In concept, the sand is first separated from the bitumen in the stripper. The substantially sand-free bitumen can then feasibly be treated with chemical and prolonged settling in the polisher to reduce water and fines contents to low levels. Bitumen lost in the splitter tails is recovered in the scrubber using a high concentration of 31 diluent. The scrubber overflow of bitumen and diluent is recycled to the stripper to conserve diluent. 32



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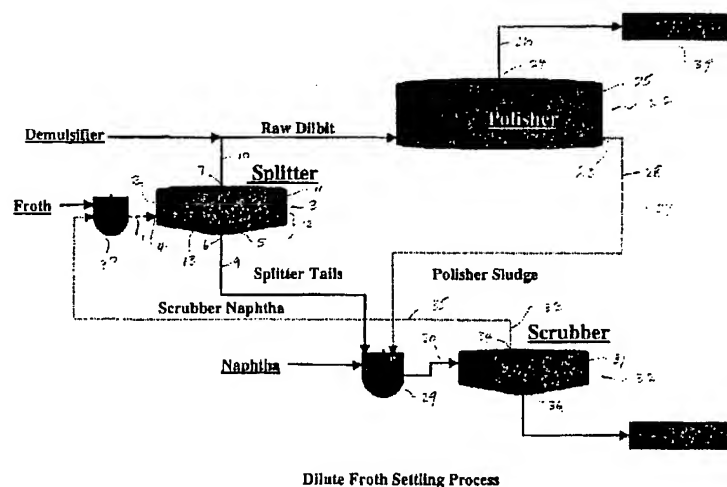
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(54) Title: STAGED SETTLING PROCESS FOR REMOVING WATER AND SOLIDS FROM OIL SAND EXTRACTION FROTH



(57) **Abrégé/Abstract:**

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Diluent-diluted bitumen froth containing bitumen and diluent hydrocarbons, water, sand and fines (collectively "dilfroth") is fed into a gravity settler ('splitter') and temporarily retained to produce a bottom layer of tails comprising sand and middlings, a rag layer of discrete three-dimensional structures, each comprising hydrocarbons contained in a skin of fines, and a top layer of hydrocarbons containing small droplets of water and fines ('raw dilbit'). The flux in the splitter is less than 6 m³/h of dilfroth fed per m² of horizontal cross-sectional rag area. The in-coming dilfroth is fed directly into the splitter middlings. Demulsifier is added to the overflow stream of raw dilbit and the mixture is subjected to prolonged settling in a polisher tank, to produce polished dilbit containing less than 1.0% water and 0.3% solids. The splitter underflow tails, containing less than 15% bitumen, is mixed with diluent to raise the diluent/bitumen ratio to 4 to 10 and is gravity settled in a scrubber. Scrubber overflow, mostly diluent containing residual bitumen stripped from the tails, is recycled to the splitter. In concept, the sand is first separated from the bitumen in the stripper. The substantially sand-free bitumen can then feasibly be treated with chemical and prolonged settling in the polisher to reduce water and fines contents to low levels. Bitumen lost in the splitter tails is recovered in the scrubber using a high concentration of diluent. The scrubber overflow of bitumen and diluent is recycled to the stripper to conserve diluent.

1 **"STAGED SETTLING PROCESS FOR REMOVING WATER AND**
2 **SOLIDS FROM OIL SAND EXTRACTION FROTH"**

3
4 **ABSTRACT OF THE DISCLOSURE**

5 Diluent-diluted bitumen froth containing bitumen and diluent
6 hydrocarbons, water, sand and fines (collectively "dilfroth") is fed into a gravity
7 settler ('splitter') and temporarily retained to produce a bottom layer of tails
8 comprising sand and middlings, a rag layer of discrete three-dimensional
9 structures, each comprising hydrocarbons contained in a skin of fines, and a
10 top layer of hydrocarbons containing small droplets of water and fines ('raw
11 dilbit'). The flux in the splitter is less than 6 m³/h of dilfroth fed per m² of
12 horizontal cross-sectional rag area. The in-coming dilfroth is fed directly into
13 the splitter middlings. Demulsifier is added to the overflow stream of raw dilbit
14 and the mixture is subjected to prolonged settling in a polisher tank, to
15 produce polished dilbit containing less than 1.0% water and 0.3% solids. The
16 splitter underflow tails, containing less than 15% bitumen, is mixed with
17 diluent to raise the diluent/bitumen ratio to 4 to 10 and is gravity settled in a
18 scrubber. Scrubber overflow, mostly diluent containing residual bitumen
19 stripped from the tails, is recycled to the splitter. In concept, the sand is first
20 separated from the bitumen in the stripper. The substantially sand-free
21 bitumen can then feasibly be treated with chemical and prolonged settling in
22 the polisher to reduce water and fines contents to low levels. Bitumen lost in
23 the splitter tails is recovered in the scrubber using a high concentration of

- 1 diluent. The scrubber overflow of bitumen and diluent is recycled to the
- 2 stripper to conserve diluent.

1 **"STAGED SETTLING PROCESS FOR REMOVING WATER AND**
2 **SOLIDS FROM OIL SAND EXTRACTION FROTH"**

3 **FIELD OF THE INVENTION**

4 The present invention relates to a gravity settling process for removing
5 contaminants, namely water and particulate solids, from diluent-diluted
6 bitumen froth derived from water-based extraction of bitumen from oil sand.

7

8 **BACKGROUND OF THE INVENTION**

9 Oil sand, as known in the Fort McMurray region of Alberta, Canada,
10 comprises water-wet, coarse sand grains having flecks of a viscous
11 hydrocarbon, known as bitumen, trapped between the sand grains. The water
12 sheaths surrounding the sand grains contain very fine clay particles. In
13 summary then, oil sand comprises: bitumen; particulate solids (coarse sand
14 and clay "fines"); and water. A sample of oil sand, for example, might
15 comprise 70% by weight sand, 14% fines, 5% water and 11% bitumen.

16 When mixed with hot water, the bitumen will separate from the sand
17 grains and be dispersed into the water phase.

18 For the past 25 years, the bitumen in McMurray oil sand has been
19 commercially recovered using a water-based process. In the first step of this
20 process, the oil sand is slurried with hot water, steam, usually some caustic
21 and naturally entrained air. The slurry is mixed, for example in a tumbler or
22 pipeline, for a prescribed retention time, to initiate a preliminary separation or
23 dispersal of the bitumen and solids and to induce air bubbles to contact and
24 aerate the bitumen. This step is referred to as "conditioning". The

1 conditioned slurry is then further diluted with hot water and introduced into a
2 large, open-topped, conical-bottomed, cylindrical vessel (termed a primary
3 separation vessel or "PSV"). The diluted slurry is retained in the PSV under
4 quiescent conditions for a prescribed retention period. During this period,
5 aerated bitumen rises and forms a froth layer, which overflows the top lip of
6 the vessel and is conveyed away in a launder. Sand grains sink and are
7 concentrated in the conical bottom. They leave the bottom of the vessel as a
8 wet tailings stream containing a small amount of bitumen. Middlings, a watery
9 mixture containing solids and bitumen, extend between the froth and sand
10 layers.

11 The wet tailings and middlings are separately withdrawn, combined
12 and sent to a secondary flotation process. This secondary flotation process is
13 commonly carried out in a deep cone vessel wherein air is sparged into the
14 vessel to assist with flotation. This vessel is referred to as the TOR vessel.
15 The bitumen recovered by flotation in the TOR vessel is recycled to the PSV.
16 The middlings from the deep cone vessel are further processed in induced air
17 flotation cells to recover contained bitumen.

18 The hot froths (80 – 85°C) produced by the PSV and flotation cells are
19 combined and subjected to cleaning, to reduce water and solids contents.

20 More particularly, it has been conventional to dilute this bitumen froth
21 with a light hydrocarbon diluent, such as a paraffinic diluent or naphtha, to
22 increase the difference in specific gravity between the bitumen and water and
23 to reduce the bitumen viscosity, to thereby aid in the separation of the water
24 and solids from the bitumen. By way of example, the composition of naphtha-

1 diluted bitumen froth typically might have a naphtha/bitumen ratio of 0.65 and
2 contain 20% water and 7% solids. (All % figures are by weight.)

3 This diluent-diluted bitumen froth, derived from water-based extraction
4 of bitumen from oil sand, is commonly referred to as "dilfroth".

5 Separation of the bitumen from water and solids is then carried out.
6 This may be done by treating the dilfroth in a sequence of scroll and disc
7 centrifuges. Alternatively, the dilfroth may be subjected to gravity separation
8 in a series of inclined plate separators ("IPS") in conjunction with
9 countercurrent solvent extraction using added light hydrocarbon diluent.

10 These prior art centrifuge and IPS techniques for removing water and
11 particulate solids from dilfroth have not been entirely satisfactory. Typically
12 the "cleaned" froth, (commonly referred to as "dilbit"), may still contain at least
13 1.5% water and 0.5 % solids. These contaminants cause problems in the
14 downstream refinery-type processes used to upgrade the dilbit to produce
15 useful end products. More particularly, the water contains chlorides, which
16 cause corrosion in heat exchangers. The solids plug catalysts. For these
17 reasons, the upgrading sector of these plants have specified that the dilbit
18 should contain <1.0% water and <0.3% solids.

19 Researchers have long sought to develop a practical and viable
20 alternative process which would reliably produce dilbit having the specified
21 smaller concentrations of water and solids. It would be even more desirable
22 to reduce the contamination to levels in the order of <0.5% water and <0.2%
23 solids. In addition, it would be desirable to achieve this using a system which
24 eliminates the centrifuges, as these are expensive to operate and cause

1 emulsification. However, solutions have been constrained by the following
2 realities:

- 3 • the clays and asphaltenes in the bitumen have an affinity for each
4 other. They tend to concentrate at water/hydrocarbon interfaces
5 and act to limit coalescence of water droplets into larger globules
6 that would settle rapidly to enable further reduction of water content
7 in the dilbit product;
- 8 • the loss of bitumen with tails must be minimal, as this is
9 environmentally undesirable and of course reduces oil recovery;
10 and
- 11 • N/B ratio in dilbit should not exceed 0.8;
- 12 • given the huge volumes processed in these operations, the
13 equipment used should be simple and reasonably inexpensive to
14 operate and additives, such as demulsifiers, should be used only
15 sparingly.

16

17 SUMMARY OF THE INVENTION

18 In accordance with the invention, the following steps are practised in
19 combination:

- 20 • Dilfroth, preferably having a diluent/bitumen ratio of 0.5 – 0.8, is fed
21 into an enclosed or vapor-tight gravity settler vessel, referred to as
22 the "splitter". The splitter is a gravity settling vessel, with outlet
23 means for withdrawal of solids and aqueous phase from the bottom
24 and outlet means for overflow of the hydrocarbon phase at the top.

1 The vessel should be enclosed at the top and vapor-tight to prevent
2 escape of diluent. The splitter has a feed inlet intermediate its
3 ends. The vessel may have a sand rake for moving sand to the
4 central bottom outlet. The dilfroth is temporarily retained (for
5 example 15 to 30 minutes) in the splitter chamber so that the froth
6 settles to form a bottom layer of sand and aqueous middlings, a rag
7 layer and a top layer of hydrocarbons (referred to as "raw dilbit").
8 Middlings is a mixture comprising mainly water containing some
9 fines and bitumen. An underflow stream of middlings and settled
10 sand, containing some hydrocarbon, (collectively referred to as
11 "splitter tails"), is removed through the bottom outlet. An overflow
12 stream of splitter raw dilbit is removed through the top outlet. The
13 splitter raw dilbit preferably comprises hydrocarbons contaminated
14 with 3 - 5% water and 0.5 - 2.5% solids. The solids are almost
15 entirely fines. The splitter tails preferably comprise mostly water
16 containing 10 - 25% solids and 8 - 20% hydrocarbons;

- 17 • In a preferred feature, the dilfroth is directly introduced into the
18 splitter middlings layer, beneath the rag layer and above the settled
19 sand. The reason for this is explained below;
- 20 • In another preferred feature, the feed rate of dilfroth to the splitter,
21 per square meter of horizontal cross-sectional rag area, is
22 maintained below 6 m³/h of dilfroth for each m² of rag area. More
23 preferably, the feed rate is maintained at about 4m³/h or less.
24 Otherwise stated, the hydrocarbons/water interface area loading

1 rate or flux is maintained below 6 m/h , preferably below 4 m/h. It is
2 found that the thickness of the rag layer begins to increase if the
3 flux is high, for example at 8 m/h. As a result, oil loss with the tails
4 increases and/or contamination of the dilbit also increases. The
5 reason for this is explained below;

- 6 • In another preferred feature, the elevation of the
7 hydrocarbon/middlings interface in the splitter chamber is
8 monitored, for example with a capacitance probe. The rate of
9 introduction of dilfroth and rate of tails removal are controlled in
10 response to the elevation of the interface, so as to maintain
11 separation of the interface from the bottom outlet by keeping the
12 interface at a generally constant elevation. This is controlled so as
13 to maintain the hydrocarbon content in the tails at less than 20%,
14 preferably less than 15%;
- 15 • In another preferred feature, the splitter raw dilbit is introduced into
16 a large vapor-tight tank (referred to as the "polisher") and
17 temporarily retained therein for a prolonged period (relative to the
18 retention time in the splitter). For example, the retention time in the
19 polisher might be in the range of 5 to 24 hours.
- 20 • In another preferred feature, demulsifier is added to the splitter raw
21 dilbit treated in the polisher. As a result of prolonged settling and
22 the use of demulsifier, water droplets coalesce and settle in the
23 polisher chamber, together with fine solids, to produce a polished
24 dilbit overhead product containing less than 1.0% water and 0.3%

1 solids, more preferably < 0.5% water and < 0.2% solids, and a
2 polisher sludge underflow comprising water and fine solids;

- 3 • In another preferred feature, the splitter tails are mixed with
4 additional diluent and settled in a vapor-tight vessel referred to as
5 the "scrubber". The scrubber is similar in structure to the splitter.
6 The scrubber diluent/bitumen ratio is quite high, preferably in the
7 range 4 to 10. At this high diluent/bitumen ratio, the diluent strips
8 residual bitumen from the splitter tails, so that there is produced a
9 scrubber overhead stream which is rich in diluent and contains most
10 of the residual bitumen. This stream is preferably recycled to the
11 splitter feed to help provide the desired splitter diluent/bitumen ratio
12 of 0.5 – 0.8. The scrubber also produces a scrubber tails underflow
13 which is mainly sand, fines and water containing less than 3%
14 bitumen.

15 With respect to the foregoing, the following will be noted:

- 16 • That the sand and most of the water originally in the dilfroth are
17 separated in the splitter and report to the splitter underflow, leaving
18 a splitter dilbit product containing fine water droplets which are
19 difficult to coalesce and separate by settling – however the volume
20 of the splitter dilbit is now considerably reduced relative to the
21 volume of the dilfroth feed. More importantly, virtually all coarse,
22 fast settling solids have been removed from the raw dilbit;
- 23 • That in the splitter hydrocarbon losses with the tails are found to be
24 <15%, preferably about 4 – 10%, of the hydrocarbons originally in

1 the dilfroth feed – in contrast, in the IPS system, between 35 – 50%
2 of the original hydrocarbons go into the tails;

3 • That in the polishing step, it is now viable to add demulsifier to the
4 splitter dilbit (reduced in volume and free of sand) and to use
5 prolonged retention time to coalesce and settle out the residual
6 water and fine solids, thereby producing a polished dilbit product
7 that meets the desired specification of less than 1.0% water and
8 0.3% solids. Because the solids entering the polisher are primarily
9 fine clays, a flat-bottom, large diameter enclosed tank can be used
10 to provide the prolonged settling (for example in the order of 5 to 24
11 hours) needed to separate the water and fines; and

12 • That in the scrubbing step, a high diluent/bitumen ratio is used to
13 scrub out residual bitumen in the splitter tails to keep bitumen
14 losses to a very low level. The added diluent is recycled
15 countercurrently to use it efficiently and to help provide the desired
16 diluent/bitumen ratio in the splitter.

17 The invention arose from a research program in which the settling
18 behaviour of dilfroth was studied using a glass-walled test circuit. Dilfroth was
19 fed into a glass column splitter through a glass inlet pipe connected with the
20 splitter between its top and bottom ends. The incoming stream of dilute froth
21 was not homogeneous. It comprised easily discernible globes of
22 hydrocarbon, pockets of muddy middlings and grains of coarse sand. As the
23 dilfroth stream entered the vessel chamber, a separation process occurred
24 due to gravity settling. As a result, a lower aqueous phase of middlings and

1 an upper hydrocarbons phase were established. The incoming dilfroth was
2 fed directly into the middlings phase. This middlings phase mainly comprised
3 a muddy suspension of clays in water. The initial separation was rapid (a few
4 seconds). The following actions were observed:

- 5 • the sand grains (60 – 150µm) settled quickly through the middlings
6 to the base of the vessel chamber. The sand and some middlings
7 were continually withdrawn and pumped to a scrubber, as further
8 described below;
- 9 • pockets of incoming middlings, containing only traces of
10 hydrocarbon, joined the aqueous phase and became part of it; and
- 11 • the incoming hydrocarbons, present in the form of discrete three
12 dimensional structures, which we referred to as "leaky sacks",
13 floated up through the aqueous phase and collected in a rag layer
14 of other oil sacks at the horizontal interface between the middlings
15 and the layer of hydrocarbons which accumulated above.

16 The leaky sacks were filled with hydrocarbons and had outer skins
17 formed of sub-micron clay particles. The composite density of the sacks was
18 lower than that of the aqueous middlings (density 1.05 – 1.1 kg/l) because
19 they would float in the middlings – but the density of the sacks was greater
20 than that of the hydrocarbon phase above the interface, because they would
21 not float in that phase (density 0.76 – 0.8 kg/l). Hence the composite density
22 of the hydrocarbon laden sacks was apparently between 0.8 and 1.05 kg/l.
23 The density of the clay alone was 2.65 kg/l.

1 The sacks formed the intermediate rag layer, approximately 100 to 200
2 mm thick at the interface, between the aqueous and hydrocarbon phases.
3 The sacks in the rag layer did not coalesce into larger ones, although some of
4 them did cluster together. They did not readily burst. They appeared to
5 crowd upwardly into the rag layer.

6 Yet the sacks did not remain in the rag layer indefinitely. They
7 appeared to penetrate the rag layer and, after residing there briefly (a minute
8 or two), they started moving downwardly through the rag layer and then sank
9 through the middlings to the bottom of the vessel chamber. This meant that
10 the sacks underwent a change in composite density and acquired a density
11 greater than that of the middlings. At the same time, the layer of
12 hydrocarbons above the rag increased in volume and excess hydrocarbon
13 overflowed the vessel. Since there was no input of hydrocarbons to the top
14 layer, other than from the sacks, and since no sacks entered the hydrocarbon
15 phase, it follows that the sacks were leaking hydrocarbons through their
16 permeable clay skins. Hence the expression "leaky sacks".

17 It is our belief that the rag layer becomes a zone of compression,
18 whereby buoyancy force from the middlings compresses the sacks against
19 the layer of hydrocarbons. As a result of this compression, hydrocarbons
20 within a sack pass through the clay skin and enter the hydrocarbon phase
21 above the rag. It is mostly the uppermost sacks in the rag layer that are
22 partially emptied of hydrocarbon by compression. These sacks increase in
23 composite density and sink to the base of the vessel chamber. However,
24 even though they are denser than the middlings, the descending sacks still

1 contain some hydrocarbons. They are only partially deflated. This is clearly
2 visible in the glass vessel, since their shape is now different. During the initial
3 floating period, a sack is spherical and full. When a sack sinks, it is thin and
4 deflated.

5 The process of hydrocarbon release from the sacks occurs only at a
6 limited rate. We refer to it as "rate of rag permeability". That is, there is only a
7 certain volume of hydrocarbon that can be released through a unit area of the
8 rag layer in a unit of time. If the delivery of new sacks to the bottom of the rag
9 layer exceeds the rate of rag permeability, the hydrocarbon release from the
10 sacks becomes the limiting factor and the process stalls gradually. The
11 process of emptying the sacks does not respond well to an increase in the
12 rate of delivery. More sacks enter the rag layer from the bottom than can be
13 emptied by the gentle compression of the layer. The depth of the rag layer
14 therefore grows. This increases the depth of rag that must be penetrated by
15 new sacks and by hydrocarbon released from them. The increased rag depth
16 also hinders the removal of partially emptied sacks from the rag layer. This
17 leads to downward rag build-up with the result that rag is withdrawn by the
18 underflow pump, causing an increase in hydrocarbons loss with the splitter
19 tails.

20 As a result of considerable experimentation, we have determined a
21 preferred limit of splitter feed rate in m^3/h for each square meter of rag
22 horizontal cross-sectional area in the splitter chamber. This flux limit is less
23 than $6 \text{ m}^3/\text{h}$ of splitter feed for each m^2 of rag area. More preferably the flux
24 should be less than about $4 \text{ m}^3/\text{h}$. At the high end of flux, the loss of

1 hydrocarbons with the splitter tails begins to increase. For example, at a flux
2 of 8 m/h the loss becomes excessive and may jeopardize the performance of
3 the scrubber. This is because the diluent/bitumen ratio in the scrubber will be
4 reduced.

5 The splitter operation does not appear to be a perfect process. Some
6 small droplets of water (a few microns in diameter) also make their way into
7 the hydrocarbon phase. The hydrocarbon layer is found to contain small
8 quantities of water (3 to 5%) and clays (1.5 to 2.5%), present in the form of
9 tiny droplets. Microscopic examination indicates that the clays are suspended
10 in water and the surfaces of water droplets are coated with clay particles. The
11 composite droplets resist coalescence and appear very stable. Removal or
12 separation of these micron sized droplets by gravity settling is very slow.
13 However, we have shown that, by prolonged settling, preferably coupled with
14 the addition of known demulsifier chemical, the composite water/clay droplets
15 can be flocculated or coalesced into much larger structures which will settle
16 out of the hydrocarbon phase over a period of hours.

17 We have also shown that a scrubbing action with a high
18 diluent/bitumen ratio (4 to 10) is effective to recover residual bitumen from the
19 splitter tails, to reduce the loss of hydrocarbons with the scrubber tails to less
20 than 1.0% bitumen and less than 6% naphtha. Most of the naphtha in
21 scrubber tails can be further recovered by steam stripping in a naphtha
22 recovery unit.

1 Broadly stated, the invention in one embodiment is a process for
2 cleaning diluent-diluted bitumen froth ("dilfroth") containing bitumen and
3 diluent hydrocarbons contaminated with water and solids, the solids mainly
4 consisting of sand and fine clay particles ("fines"), comprising: providing a
5 splitter vessel forming a vapor-tight chamber for gravity settling, said vessel
6 having an overflow outlet at its upper end, an underflow outlet at its lower end
7 and means for feeding incoming dilfroth into the chamber; feeding dilfroth into
8 the chamber through the feed means and temporarily retaining it therein so
9 that the froth settles to form a bottom layer of tails comprising aqueous
10 middlings and substantially all of the sand, said tails containing some
11 hydrocarbons, an intermediate layer of rag comprising water, fines and
12 hydrocarbons collected in discrete three dimensional structures, and a top
13 layer of raw dilbit comprising mainly hydrocarbons containing some water and
14 fines, said middlings combining with the rag and dilbit to create a discernible
15 hydrocarbons/water interface; the feed means being operative to directly feed
16 the incoming dilfroth into the middlings; removing dilbit through the overflow
17 outlet; and removing tails through the underflow outlet, said tails containing
18 less than 20% of the hydrocarbons in the froth.

19 Broadly stated, in another embodiment the invention is a process for
20 cleaning diluent-diluted bitumen froth ("dilfroth") containing bitumen and
21 diluent hydrocarbons contaminated with water and solids, the solids mainly
22 consisting of sand and fine clay particles ("fines"), comprising: subjecting the
23 dilfroth to gravity settling in a vapor-tight first zone of separation to produce an
24 overflow stream of raw dilbit, comprising hydrocarbons containing less than

1 5% water and 2% fines, and an underflow stream of tails comprising aqueous
2 middlings and substantially all of the sand, said tails containing less than 15%
3 hydrocarbons; and subjecting the raw dilbit (preferably mixed with a small
4 addition of suitable chemical) to gravity settling in an enclosed second zone of
5 separation for sufficient time to produce an overflow stream of polished dilbit
6 containing less than 1.0% water and 0.3% solids and an underflow stream of
7 polisher sludge.

8

9

DESCRIPTION OF THE DRAWINGS

10 Figure 1 is a schematic showing the vessels and steps of the process;

11 Figure 2a is a schematic showing the laboratory pilot circuit, in
12 polishing configuration, as used to develop the data of Example I;

13 Figure 2b is a schematic showing the laboratory pilot circuit, in
14 scrubbing configuration, as used to develop the data of Example II;

15 Figure 3 is a plot showing dilbit water content when settled over time
16 when the dilbit does not contain demulsifier; and

17 Figure 4 is a plot showing dilbit water content when settled over time
18 when the dilbit contains demulsifier.

19

20

DESCRIPTION OF THE PREFERRED EMBODIMENT

21 The invention is concerned with a process for cleaning diluent-diluted
22 bitumen froth by reducing the content of contaminants, specifically water and
23 solids.

1 Bitumen froth is initially received from a plant (not shown) for extracting
2 bitumen from oil sand using the known hot water process. The froth, as
3 received, is at elevated temperature (for example 85°C) and typically
4 comprises:

5	bitumen -	60%
6	water -	30%
7	solids -	10%

8 A light hydrocarbon diluent, such as process naphtha, is mixed with the
9 froth in a mixer 1 to provide diluent-diluted bitumen froth. The naphtha is at
10 least partly supplied by recycling scrubber naphtha, produced as described
11 below.

12 The scrubber naphtha is supplied in an amount such that the
13 naphtha/bitumen ratio of the diluent-diluted froth ("dilfroth") is preferably in the
14 range 0.5 – 0.8, most preferably about 0.65.

15 The dilfroth 38 is fed by line 1 from the mixer 37 into the chamber 2 of
16 a gravity settler vessel, referred to as the "splitter" 3. The dilfroth 38 is fed into
17 the chamber 2 through inlet means 4. The splitter 3 has a conical bottom 5. It
18 has underflow and overflow outlets 6, 7 at its bottom and top ends,
19 respectively. A pump 8 and line 9 withdraw a stream of splitter tails 13
20 through the underflow outlet 6. Splitter overflow line 10 collects an overflow
21 stream of raw dilbit.

22 The rate at which dilfroth 38 is fed to the splitter chamber 2 and the
23 diameter of the cylindrical section 11 of the splitter 3 are selected to ensure a
24 preferred flux of < 6 m/h, most preferably about 4 m/h.

1 The dilfroth 38 is temporarily retained in the splitter chamber 2 for
2 sufficient time so that gravity settling takes place to produce the following:

- 3 • a bottom layer 12 of splitter tails 13, comprising mainly sand 14 and
4 aqueous middlings 15, said tails containing some hydrocarbons;
5 • an intermediate layer 16 of rag 17, said rag comprising mainly
6 hydrocarbons associated with water and fines in discrete three
7 dimensional structures or sacks 18; and
8 • a top layer 19 of raw dilbit 20 comprising mainly hydrocarbons
9 containing some water and fines (clay particles).

10 The middlings 15 combine with the rag 17 and raw dilbit 20 to create a
11 discernible hydrocarbons/water interface 21.

12 The splitter inlet means 6 delivers incoming dilfroth 38 into the
13 middlings 15 across the cross-section of the splitter chamber 2, at an
14 elevation spaced below the layer of rag 17 and well above the underflow
15 outlet 6.

16 Means, such as a capacitance probe, may be used to monitor the
17 elevation of the hydrocarbons/water interface 21. The rates of feeding dilfroth
18 38 and withdrawing tails 13 may then be controlled in response to the probe
19 readings to maintain the elevation of the interface 21 generally constant. It is
20 of course desirable to keep the interface 21 away from the bottom of the
21 splitter chamber 2, to minimize hydrocarbon losses with the splitter tails 13.
22 Alternatively one may monitor the composition of the splitter tails 13 and vary
23 the rates with the objective of keeping the tails hydrocarbon content below a
24 predetermined value, usually less than 15%.

1 The raw dilbit 20 produced through the splitter overflow outlet 7 is
2 pumped through line 10 to a flat-bottomed, vapor-tight tank, referred to as the
3 "polisher" 22, and subjected to gravity settling therein. Preferably a
4 demulsifier is added to the raw dilbit 20 as it moves through the line 10. For
5 example, 40 ppm of Champion MR 121-6 demulsifier may be added for this
6 purpose.

7 The polisher 22 has a bottom underflow outlet 23 and a top overflow
8 outlet 24.

9 The raw dilbit/demulsifier mixture is temporarily retained for a
10 prolonged period (for example, 24 hours) in the polisher chamber 25. Water
11 droplets coalesce and settle, together with fines. Polished dilbit 39 is
12 removed as an overflow stream from the polisher 22 through line 26. The
13 polished dilbit 39 is found to comprise hydrocarbons containing < 1.0% water
14 and < 0.3% solids. Polisher sludge 27, comprising water, solids and less than
15 15% hydrocarbons, is removed from the polisher 22 as an underflow stream
16 through line 28. It is pumped through line 28 into scrubber mixer 29.

17 The splitter tails 13 produced through the splitter underflow outlet 6 are
18 also pumped through line 9 to scrubber mixer 29. Naphtha is added to the
19 splitter tails 13 and polisher sludge 27 in the scrubber mixer 29 to produce a
20 scrubber feed 30 preferably having a naphtha/bitumen ratio in the range 4 to
21 10, more preferably 5 to 8. The scrubber feed 30 is introduced into the
22 chamber 31 of a vapor-tight vessel, referred to as the "scrubber" 32. The
23 scrubber feed 30 is temporarily retained in the scrubber chamber 31 (for
24 example for 20 to 30 minutes) and subjected to gravity settling therein. A

1 scrubber overflow stream 33 of hydrocarbons, mainly comprising naphtha
2 associated with some bitumen, is removed through an overflow outlet 34 and
3 recycled through line 35 to splitter mixer 37. A scrubber underflow stream of
4 scrubber tails 36, comprising water and solids containing some hydrocarbons,
5 is removed and forwarded to a naphtha recovery unit (not shown).

6 The nature and utility of the process is demonstrated by the following
7 examples.

8

9 **Example 1 – Splitter and Polisher**

10 This example demonstrates the results obtained when a splitter and
11 polisher were operated together in series.

12 In this experiment, two glass columns were supplied as splitter and
13 polisher and connected as shown in Figure 2a. Hot bituminous froth and
14 naphtha were combined in an agitated mixer 37, at naphtha/bitumen ratios of
15 0.55/1.0 by weight. The mixture was pumped continuously, at a rate of 191
16 g/min, to the splitter column 3 (ID- 125 mm and height – 750 mm). The
17 calculated splitter flux was 0.93 m/h.

18 The separation process was clearly visible through the glass walls of
19 the splitter column, with coarse sand settling and hydrocarbon sacks 40
20 floating. The full sacks 40 accumulated at the aqueous/hydrocarbon interface
21 21 and a deep layer 19 of hydrocarbon formed above the interface. After
22 residing at the interface for a few minutes, partially emptied sacks 41 of
23 hydrocarbon were observed sinking and joining the settled solids or splitter
24 tails 13 at the bottom. The rate of the splitter tails stream withdrawal through

1 line 9 was adjusted manually to maintain the level of aqueous/hydrocarbon
2 interface 21 a few inches above the feed injection point 42.

3 As shown in Table 1, the splitter overflow or raw dilbit 20 contained
4 about 4.57% water and 0.76 % solids.

5 The splitter overflow was continuously fed through line 10 into the
6 polisher 22, where the residence time was about 35 min. During this short
7 residence time, water content in the polisher overflow (polished dilbit) dropped
8 to 2.91 % and solids content down to 0.36%. This polished dilbit quality would
9 not be adequate for further processing in the bitumen upgrading plant.

10 Table 1
11 Summary of continuous splitter/polisher operation without demulsifier

	Splitter Feed	Splitter Overflow	Splitter Underflow	Polisher Overflow	Polisher Underflow
Flow Rate, mg/l	191.14	145.96	45.18	132.29	13.67
Bitumen, %	48.16	59.72	10.81	60.02	56.71
Naphtha, %	26.57	32.65	6.92	34.36	16.04
Water, %	19.97	4.57	69.68	2.91	20.72
Solids	4.77	0.76	17.75	0.36	4.58

12
13 To improve dilbit quality, a series of experiments were conducted to
14 investigate the effects of demulsifier addition and prolonged settling. In one
15 such experiment, demulsifier was continuously injected into the splitter
16 overflow, at a dosage of 40 ppm, before it entered the polisher column. When
17 the polisher column was completely filled with the demulsifier-treated raw

1 dilbit, the operation was stopped. At this point, both the splitter and polisher
2 columns were completely filled with dilbit. However, the first splitter column
3 contained dilbit with no demulsifier and the polisher column contained dilbit
4 with 40 ppm of demulsifier. The diluted bitumen in the two columns was
5 allowed to stand for up to 26 h while the temperature inside the columns was
6 controlled at 80°C, by re-circulating hot water through water jackets. Samples
7 from different depths of the two vessels were taken periodically for water
8 content analysis by Karl Fisher titration.

9 Figure 3 shows the water contents in diluted bitumen remaining in the
10 first column, as a function of settling time. As illustrated, the water content
11 dropped from 3.5% to 2.8% in the first hour and to 2.4% within 3 hours. After
12 settling for 6 hours, the water content stayed at a constant level, 2.2%.
13 Further increases in settling time, up to 25 h, did not change the water content
14 at all. This indicates that the final concentration of 2.2% was very stable.
15 Without demulsifier, it was not possible to remove the remainder of water by
16 gravitational settling within a reasonable settling time.

17 However, in the second column, where 40 ppm of demulsifier were
18 present in the dilbit, the removal of water improved dramatically, as shown in
19 Figure 4. Water content dropped from 3.5% to 1.3 %, even during the
20 continuing operation, when the second column was filling up (about 30 min).
21 After 3 hours of settling, water content dropped to 0.8% and eventually down
22 to 0.2% over the 26 hour period. As demonstrated, the addition of demulsifier
23 to the polisher feed, combined with extended settling, can produce essentially
24 dry dilbit.

1 Figures 3 and 4 also show that the water contents at different depths
2 are almost the same, in the range of depths studied in this work. This reveals
3 that the water separation is not a simple droplet settling process, where
4 settling velocity is controlled by the size of droplets as introduced in the feed.
5 Based on the test results, we postulate that the demulsifier de-stabilizes the
6 water emulsion and allows small droplets to form much larger aggregates.
7 Once the droplets agglomerate to larger formations, they settle out very
8 quickly through the entire depth of the oil phase. The residence time, required
9 in the polisher, is the time needed for the process of droplet agglomeration to
10 be completed.

11 In summary, the splitter overflow raw dilbit advantageously can be
12 treated by the addition of suitable demulsifier and settling in a separate vessel
13 for sufficient time to achieve the desired level of water content. With addition
14 of appropriate demulsifier and given sufficient settling time (at least 12 h), a
15 very high quality dilbit can be produced.

16

17 **Example 2 – Splitter and Scrubber**

18 In another experiment, the same two columns were connected as
19 shown in Figure 2b and operated as splitter and scrubber. Bituminous froth
20 was initially mixed with fresh naphtha in the feed mixer 37 and introduced to
21 the splitter column as described in Example 1. The separation process in the
22 splitter column was the same as in experiment 1. The splitter overflow stream
23 was weighed and sampled. The tailings stream from the splitter was mixed
24 with fresh naphtha, added at a rate of 64.35 g/minute, and introduced to the

1 scrubber column. Since the amount of bitumen remaining in splitter tails was
 2 only a small fraction of the total hydrocarbon ("HC") entering the process, the
 3 N/B ratio in the scrubber was an order of magnitude higher than that in the
 4 splitter feed. As previously explained, it was determined that at N/B ratios
 5 exceeding 4/1, the HC/clay structures responsible for the formation of the rag
 6 become unstable.

7 The released HC in the scrubber overflow contained very little water
 8 and solids, whereas the scrubber underflow tails contained very little
 9 hydrocarbon. As soon as the scrubber overflow stream became available for
 10 re-circulation, we discontinued the addition of fresh naphtha to the fresh froth
 11 and replaced it with the high N/B scrubber overflow. The process continued
 12 for four hours, until we were satisfied that a steady flow condition was
 13 reached. The material balance data, recorded during the steady state
 14 condition, are shown in Table 2.

15 Table 2

16 Summary of continuous splitter/scrubber operation without demulsifier

	Splitter Feed	Splitter Overflow	Splitter Underflow	Scrubber Feed	Scrubber Overflow	Scrubber Underflow
Flow Rate, mg/l	259.59	183.55	76.03	140.38	80.60	59.78
Bitumen, %	46.57	57.77	19.51	10.56	17.10	1.75
Naphtha, %	30.05	37.69	11.59	51.97	89.95	0.78
Water, %	18.08	3.57	53.09	28.76	1.09	66.06
Solids, %	3.48	0.68	10.23	5.54	0.06	27.48

17

- 1 Bitumen and naphtha recoveries in this experiment were 99.43% and 99.14%
- 2 respectively.
- 3

1 THE EMBODIMENTS OF THE INVENTION IN WHICH AN
2 EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS
3 FOLLOWS:

4 1. A process for cleaning diluent-diluted bitumen froth ("dilfroth")
5 containing bitumen and diluent hydrocarbons contaminated with water and
6 solids, the solids mainly consisting of sand and fine clay particles ("fines"),
7 comprising:

8 providing a splitter vessel forming a vapor-tight chamber for gravity
9 settling, said vessel having an overflow outlet at its upper end, an underflow
10 outlet at its lower end and means for feeding incoming dilfroth into the
11 chamber;

12 feeding dilfroth into the chamber through the feed means and
13 temporarily retaining it therein so that the froth settles to form a bottom layer
14 of tails comprising aqueous middlings and substantially all of the sand, said
15 tails containing some hydrocarbons, an intermediate layer of rag comprising
16 water, fines and hydrocarbons collected in discrete three dimensional
17 structures, and a top layer of raw dilbit comprising mainly hydrocarbons
18 containing some water and fines, said middlings combining with the rag and
19 dilbit to create a discernible hydrocarbons/water interface;

20 the feed means being operative to directly feed the incoming dilfroth
21 into the middlings;

22 removing dilbit through the overflow outlet; and

23 removing tails through the underflow outlet, said tails containing less
24 than 20% of the hydrocarbons in the froth.

1 2. The process as set forth in claim 1 wherein the rate of feeding
2 incoming froth is maintained at less than $6 \text{ m}^3/\text{h}$ for each m^2 of horizontal
3 cross-sectional rag area.

4

5 3. The process as set forth in claim 1 comprising:
6 monitoring the hydrocarbons/water interface and controlling at least
7 one of feeding and removing rates to maintain the interface at a generally
8 constant elevation to limit hydrocarbon losses with the tails.

9

10 4. The process as set forth in claim 2 comprising:
11 monitoring the hydrocarbons/water interface and controlling feeding or
12 removing rates to maintain the interface at a generally constant elevation to
13 limit hydrocarbon losses with the tails.

14

15 5. The process as set forth in claim 1 wherein the dilbit contains less
16 than 3% solids and less than 8% water.

17

18 6. The process as set forth in claim 2 wherein the dilbit contains less
19 than 3% solids and less than 8% water.

20

21 7. The process as set forth in claim 3 wherein the dilbit contains less
22 than 3% solids and less than 8% water.

1 8. The process as set forth in claim 4 wherein the dilbit contains less
2 than 3% solids and less than 8% water.

3

4 9. The process as set forth in claim 1, 2, 3, 4, 5, 6, 7 or 8 wherein:
5 the diluent/bitumen ratio in the froth is in the range 0.5 – 0.8.

6

7 10. A process for cleaning diluent-diluted bitumen froth ("dilfroth")
8 containing bitumen and diluent hydrocarbons contaminated with water and
9 solids, the solids mainly consisting of sand and fine clay particles ("fines"),
10 comprising:

11 subjecting the froth to gravity settling in an enclosed first zone of
12 separation to produce an overflow stream of raw dilbit, comprising mainly
13 hydrocarbons containing small amounts of water and fines, and an underflow
14 stream of tails, comprising aqueous middlings and substantially all of the
15 sand, said tails containing less than 20% hydrocarbons; and

16 subjecting the raw dilbit to gravity settling in an enclosed second zone
17 of separation for sufficient time to produce an overflow stream of polished
18 dilbit containing less than 1.0% water and less than 0.3% solids and an
19 underflow stream of polisher sludge.

20

21 11. The process as set forth in claim 10 comprising:

22 adding demulsifier to the raw dilbit treated in the second zone of
23 separation.

24

1 12. The process as set forth in claim 10 wherein:

2 the raw dilbit contains less than 3% solids and less than 8% water and
3 the tails contains less than 15% hydrocarbons.

4

5 13. The process as set forth in claim 11 wherein:

6 the raw dilbit contains less than 3% solids and less than 8% water and
7 the tails contains less than 15% hydrocarbons.

8

9 14. The process as set forth in claim 10 wherein:

10 the rate of feeding incoming froth is maintained at less than $6 \text{ m}^3/\text{h}$ for
11 each m^2 of horizontal cross-sectional rag area.

12

13 15. The process as set forth in claim 11 wherein:

14 the rate of feeding incoming froth is maintained at less than $6 \text{ m}^3/\text{h}$ for
15 each m^2 of horizontal cross-sectional rag area.

16

17 16. The process as set forth in claim 12 wherein:

18 the rate of feeding incoming froth is maintained at less than $6 \text{ m}^3/\text{h}$ for
19 each m^2 of horizontal cross-sectional rag area.

20

21 17. The process as set forth in claim 13 wherein:

22 the rate of feeding incoming froth is maintained at less than $6 \text{ m}^3/\text{h}$ for
23 each m^2 of horizontal cross-sectional rag area.

24

1 18. The process as set forth in claims 10, 11, 12, 13, 14, 15, 16 or 17
2 comprising:

3 mixing the first zone tails with diluent and subjecting the produced
4 diluted tails to gravity settling in an enclosed third zone of separation to
5 produce an overhead stream of scrubber hydrocarbons and an underflow
6 stream of scrubber tails; and

7 recycling scrubber hydrocarbons to the first zone.

8

9 19. The process as set forth in claims 11, 12, 13, 14, 15, 16 or 17
10 comprising:

11 diluting the first zone tails with diluent and subjecting the diluted tails to
12 gravity settling in an enclosed third zone of separation to produce an
13 overhead stream of scrubber hydrocarbons and an underflow stream of
14 scrubber tails;

15 recycling scrubber hydrocarbons to the first zone; and wherein

16 a vapor-tight splitter vessel chamber provides the first zone of
17 separation;

18 a vapor-tight polisher vessel chamber provides the second zone of
19 separation; and

20 a vapor-tight scrubber vessel chamber provides the third zone of
21 separation.

22

23 20. The process as set forth in claims 10, 11, 12, 13, 14, 15, 16 or 17
24 comprising:

1 diluting the first zone tails with diluent and subjecting the diluted tails to
2 gravity settling in an enclosed third zone of separation to produce an
3 overhead stream of scrubber hydrocarbons and an underflow stream of
4 scrubber tails;

5 recycling scrubber hydrocarbons to the first zone; and wherein

6 a vapor-tight splitter vessel chamber provides the first zone of
7 separation;

8 a vapor-tight polisher vessel chamber provides the second zone of
9 separation; and

10 a vapor-tight scrubber vessel chamber provides the third zone of
11 separation;

12 monitoring the hydrocarbons/water interface and controlling at least
13 one of feeding and removing rates to maintain the interface at a generally
14 constant elevation to limit hydrocarbon losses with the tails.

15

16 21. The process as set forth in claims 10, 11, 12, 13, 14, 15, 16 or 17
17 comprising:

18 diluting the first zone tails with diluent and subjecting the diluted tails to
19 gravity settling in an enclosed third zone of separation to produce an
20 overhead stream of scrubber hydrocarbons and an underflow stream of
21 scrubber tails;

22 recycling scrubber hydrocarbons to the first zone; and wherein

23 a vapor-tight splitter vessel chamber provides the first zone of
24 separation;

1 a vapor-tight polisher vessel chamber provides the second zone of
2 separation; and

3 a vapor-tight scrubber vessel chamber provides the third zone of
4 separation;

5 monitoring the hydrocarbons/water interface and controlling at least
6 one of feeding and removing rates to maintain the interface at a generally
7 constant elevation to limit hydrocarbon losses with the tails; and

8 sufficient diluent is added to the first zone tails and sufficient scrubber
9 hydrocarbons are recycled to maintain the diluent/bitumen ratio in the first
10 zone I the range 0.5 to 0.8.

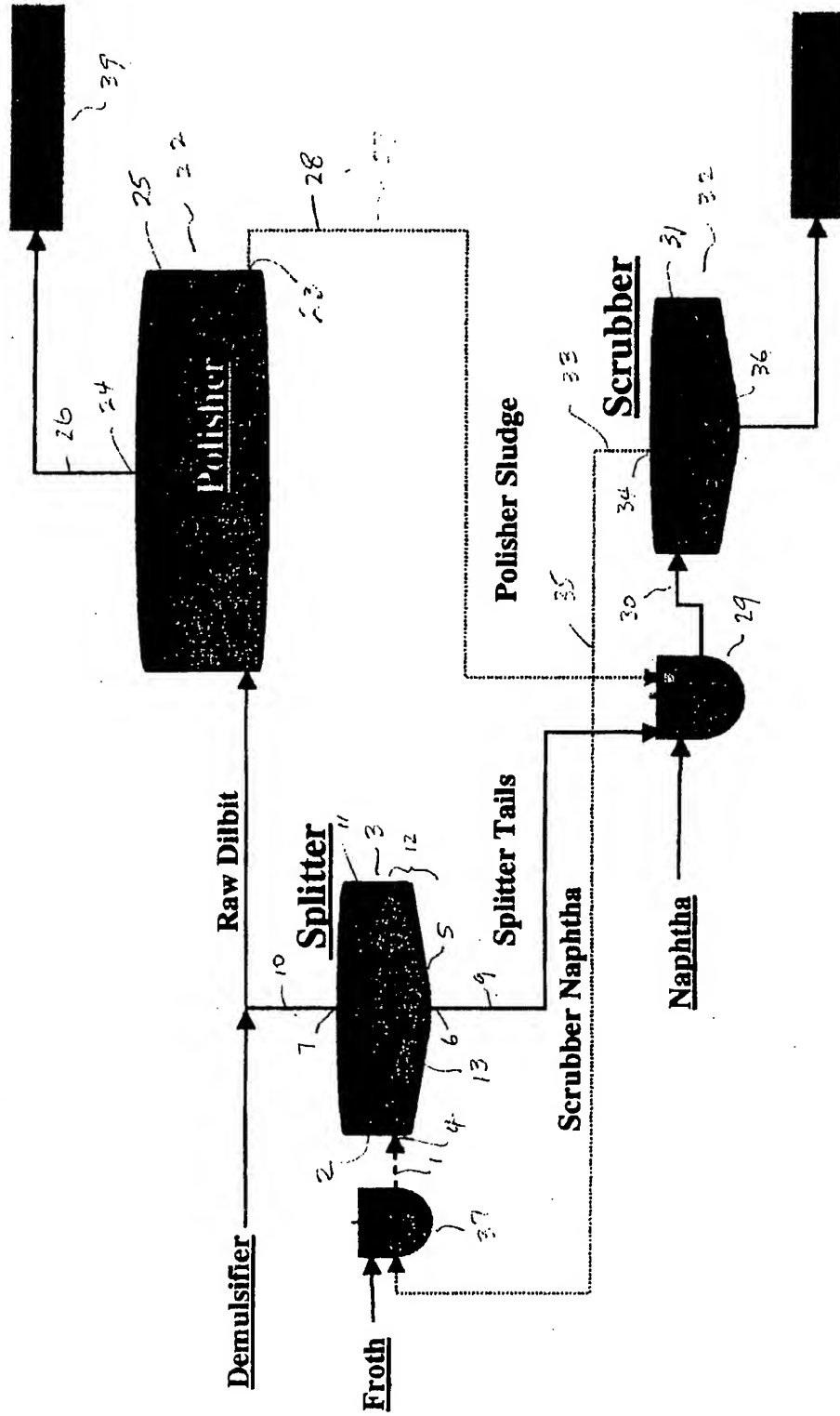


Figure 1. Dilute Froth Settling Process

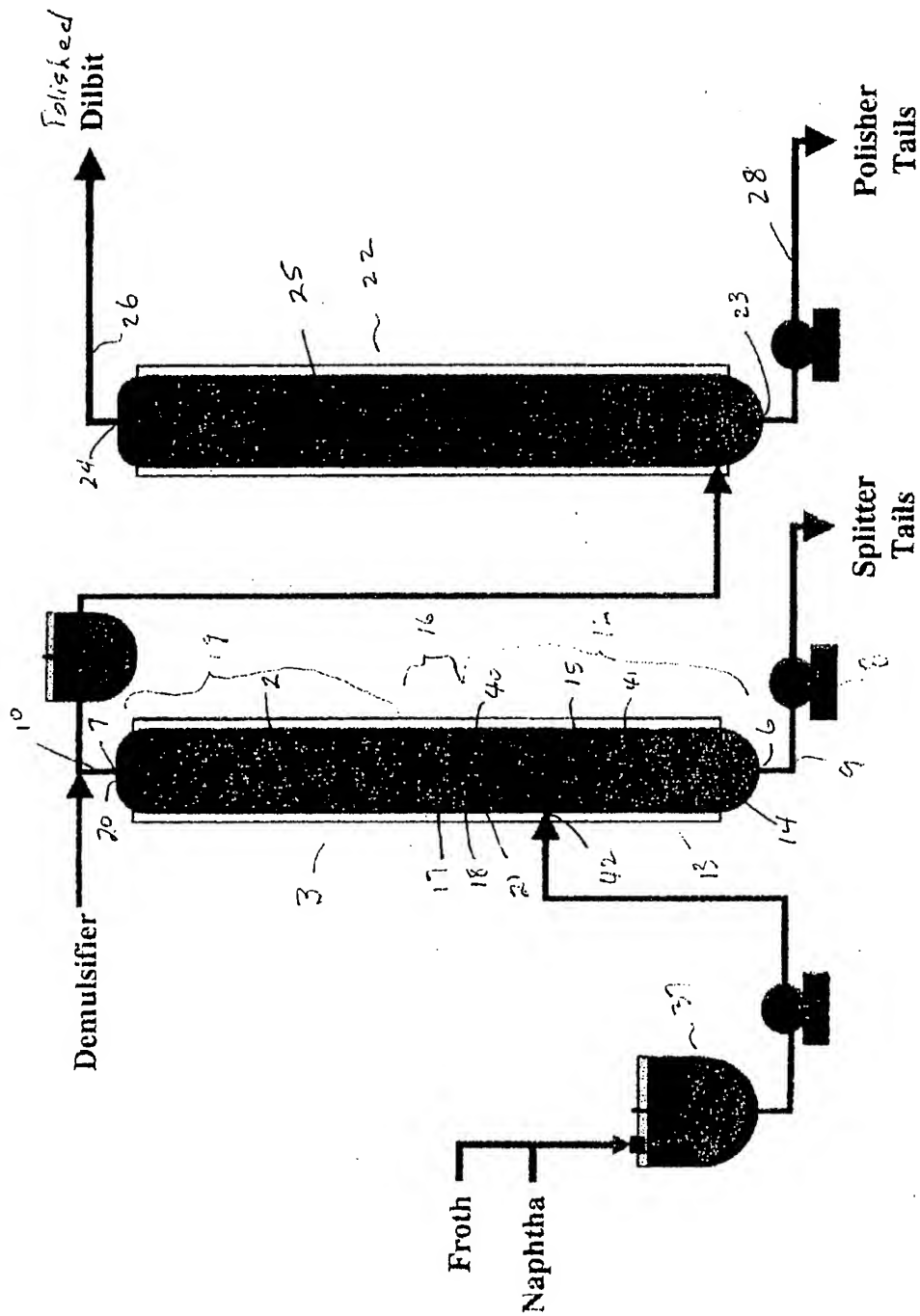


Figure 2a. Simplified Laboratory Set-up in Polishing Configuration

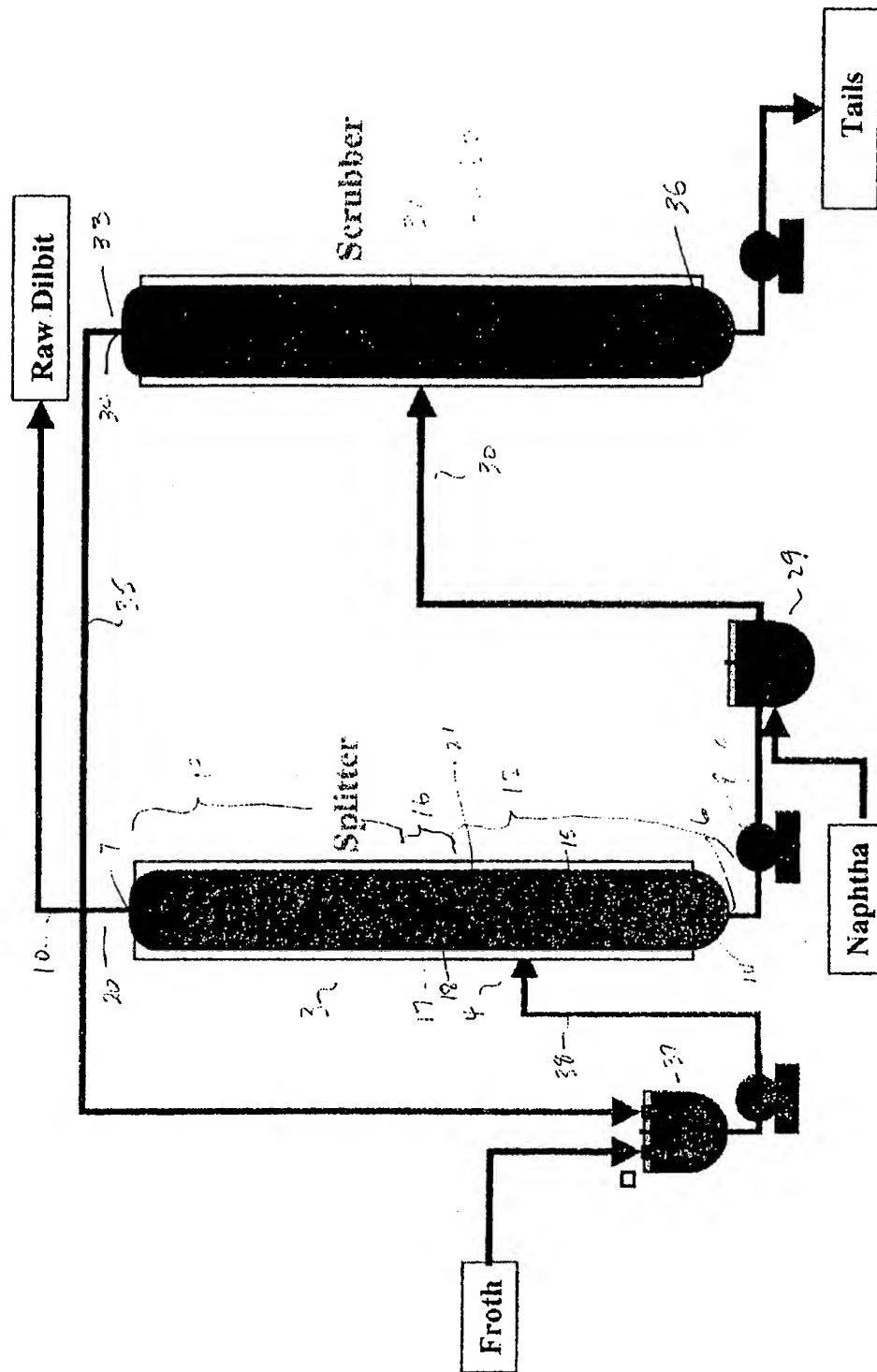
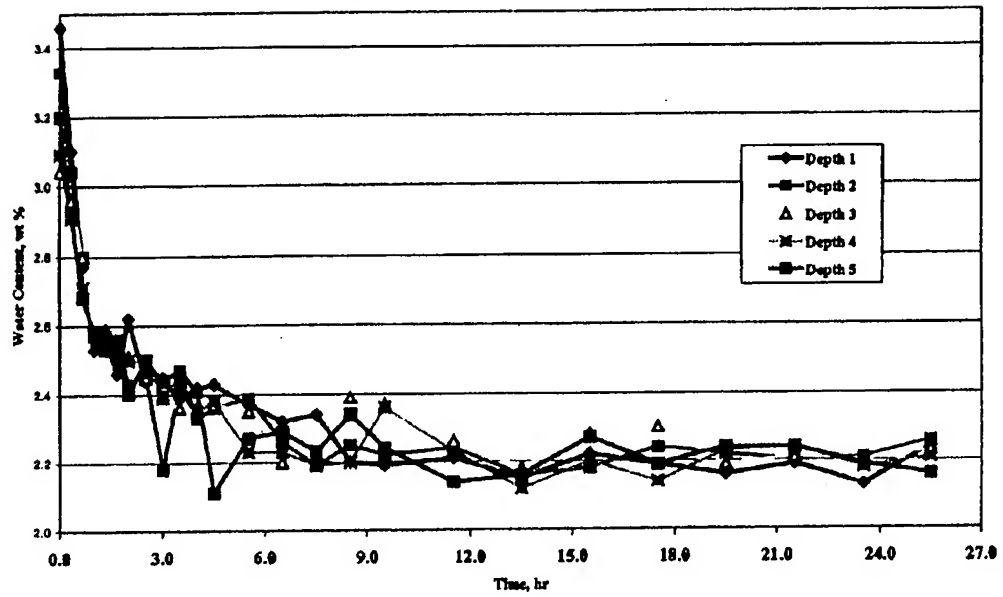


Figure 2b. Simplified Laboratory Setup in Tailings Scrubber Configuration

3
 Figure 2. Dilute Bitumen Dehydration by Settling
 80 Deg. C, N/B - 0.65, No Demulsifier



4
 Figure 3. Dilute Bitumen Dehydration by Settling
 80 Deg. C, N/B - 0.65, 40 ppm Champion MR 121-6

